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CA

Also dyes from 1,5-anthracenaphthol and its derivatives.
VII. Titration of intermediate products and also dyes of
the naphthalene series. V. V. Perchalin (Lensoviet Inst.
Technol., Leningrad). *J. Gen. Chem. U.S.S.R.* 21, 1157-1160
(1951) (Engl. translation).—See *C.A.* 45, 10008a. B. R.

CA

Also dyes from 1,5-aminonaphthol and some of its derivatives. VIII. The absorption spectra of some intermediates and one dye of the naphthalene series. V. V. Parshala and M. V. Savits'yanova (Leningrad Technol. Inst., Leningrad). *Zhur. Obshchei Khim.* (J. Gen. Chem.) 21, 1339-50 (1951); cf. C.A. 45, 10006c. —The spectra of the naphthalene compounds studied here show, with a few deviations, the bands of the naphthalene mol., while the dyes display new specific bands. The ortho-OH and para-OH compounds exhibit intramolecular H-bonding. 1,5-Aminonaphthol shows max. at 227 mμ, and 315 mμ. Its 2-sulfonic acid shows a max. at 228 and 340 mμ. The 4-sulfonic acid has max. at 227 and 340 mμ, while the 6-sulfonic acid has max. at 227 and 340 mμ. 1,5-Aminonaphthol-7-sulfonic acid has max. at 223, 225, and 342 mμ. 1-Benzeneazo-1-naphthol has max. at 223, 270, and 370 mμ; its HCl salt has max. at 230, 280, 380, and 540 mμ. 4-Benzeneazo-1-naphthylamine has max. at 230, 280, 380, and 435 mμ, and its HCl salt at 223, 230, 270, 370, and 550 mμ. 2-Benzeneazo-1-naphthol has max. at 235, 295, 365, and 490 mμ. Its 4-benzeneazo analog has max. at 208, 290, and 490 mμ. 2-Benzeneazo-1,5-aminonaphthol has max. at 230, 300, and

505 mμ, while its 3-benzeneazo analog has max. at 208, 295, 305, and 530 mμ. The absorption max. of the following in EtOH and EtOH-NaOH, resp., are: 2-(p-hydroxybenzene-1-naphthol): 480 and 490; 4-(p-hydroxybenzene-1-naphthol): 480 and 490; 2-(p-aminobenzeneazo)-1,5-aminonaphthol: 510 and 505; 2-(p-hydroxybenzeneazo)-1,5-aminonaphthol: 500 and 500; 1-(p-hydroxybenzeneazo)-2,8-aminonaphthol: 505 and 530; 1,5-bis(benzeneazo)-2,8-aminonaphthol: 505 and 540 mμ. 4-Benzeneazo-1,5-aminonaphthol-3-sulfonic acid has abs. max. 237, 270, and 500; 6-benzeneazo analog: 230, 290, 360, and 530; 2-benzeneazo-1,5-aminonaphthol-4-sulfonic acid: 235, 310, and 460; 6-benzeneazo-1,5-aminonaphthol-4-sulfonic acid: 235, 310, 345, and 580; aminonaphthol-4-sulfonic acid: 230, 310; 3-benzeneazo-1,5-aminonaphthol-7-sulfonic acid: 227, 320, 370, and 515; and 510; 6-benzeneazo analog: 227, 320, 370, and 515; 3-benzeneazo-1,5-aminonaphthol-6-sulfonic acid: 219, 290, 360, and 530; 6-benzeneazo analog: 222, 290, 360, and 530; 2,8-bis(benzeneazo)-1,5-aminonaphthol-6-sulfonic acid: 215, 350, 360, and 540; 3-benzeneazo-1,5-aminonaphthol-8-sulfonic acid: 208, 325, and 520; 6-benzeneazo-1,5-aminonaphthol-8-sulfonic acid: 234, 320, and 510; and 2,8-bis(benzeneazo)-1,5-aminonaphthol-8-sulfonic acid: 240, 325, and 540 mμ, resp.

G. M. Kozlovskii

PEREKALIN, V. V.

"Reactions of diketenes. Interaction of diketenes with some aromatic amines of low basicity." Perekalin, V. V. and Lerner, O. M. (p. 1995)

SO: Journal of General Chemistry (Zhurnal Obshchei Khimii) 1951, Vol 21, No 11.

V. V. PEREKALIN

Nov 51

USSR/Chemistry - Diketene

Nov 51

"Interaction of Diketene with Certain Weakly
Basic Aromatic Amines," V. V. Perekalin, O. M.

Kerner

"Zhur Opshch Khim" Vol XXI, No 11, pp 1995-2001

Shown for the 1st time that diketene can be used
successfully to prep acetoacetyl derivs of weakly
basic amines. Prep'd acetoacetyl derivs of o-
nitroaniline and diphenylamine with much higher
yields than by other methods. Shown feasibility
of use of acetoacetyl derivs as azotols and for
prep of complex lepidone derivs. Shown that

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Nov 51

USSR/Chemistry - Diketene (Contd)

acetoacetic acid chloride can be used to acylate
amphoteric amines (carbazole, etc). Proposed
mechanism of activating action of pyridine on
diketene.

194746

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Absorption spectra of some derivatives of di- and tri-phenylmethane series. V. V. Pivkalkin, M. V. Serov, Yanova, and R. I. Mironova. *Zhur. Obshch. Khim.* (J. Gen. Chem.) 23, 881-9 (1959).—Spectra of numerous deriva. of di- and triphenylmethane series indicate the presence in the spectra of the derived dyes of abs. bands of the starting materials, with a shift of the position of the shorter-wave bands and with a redistribution of their intensities. EtOH was used as solvent and the pH of solutions was 8.0. The following abs. bands (mμ) and extinction coeffs. ($\times 10^{-4}$) were observed: PhNMe₂, 251(1.34), 290(0.53); (*p*-Me₂NC₆H₄)₂CH₂, 215(2.6), 262(2), 300(0.42); (*p*-Me₂NC₆H₄)₂CHPh, 260(0.73), 300(0.094); (*p*-Me₂NC₆H₄)₂CHCHPh, 260(3.47), 262(3.82), 268(3.2), 300(0.53); (*p*-Me₂NC₆H₄)₂CHCH(OH)NMe₂, 267(2.48), 268(1.4), 264(1.58), 300(0.74); (*p*-Me₂NC₆H₄)₂CHCH(OH)Ph, 267(1.4), 264(1.58), 300(0.74); (*p*-Me₂NC₆H₄)₂COH, 260(0.24), 300(0.11). The pos. ion of the colored salts of bis(*p*-dimethylaminophenyl)-methane (from the respective carbinol) gives in EtOH, at pH 8, abs. max. at 263(1.14), 300(0.52), 340(0.058), and 454(0.03); Malachite Green (in buffer soln, pH 4.2) gave 249(0.35), 316.5(0.82), 425(0.86), and 620(3.44), while

crystal violet gave (in buffer pH 6.07) 250(1.09), 305(1.37), 360(0.13), and 600(6.97). In concd. HCl Malachite Green shows the disappearance of only the longer-wave band, while the other 3 remain unchanged; crystal violet behaves similarly and also loses the 360 band, with appearance of a new band at 435 mμ. Univalent pos. ion of Malachite Green gives 249(0.35), 316.5(0.82), 425(0.86), and 620(3.44); bivalent ion gives 200(0.25), 320(0.12), and 435(0.275); while trivalent pos. ion of crystal violet gives 250(0.99), 280(0.7), 314(0.375), 436(0.98) (the latter 2 in 30% HCl), while the univalent pos. ion of *p*-dimethylaminocarbonylmethane (from the carbinol; run in dil. HCl), gave 250(—), 280(—), 330(—), and 450(—). Phenylmethylpyrazolone has one band at 246 mμ and its colored deriva. retain this band, with addn. of longer-wave bands.

G. M. Kosolapov

PEREKALIN, V. V.
USSR/Chemistry - Physical chemistry

Card 1/2 Pub. 43 - 47/62

Authors : Perekalin, V. V.

Title : Hydrogen bond and the structure of aromatic compounds

Periodical : Izv. ANSSSR. Ser. fiz. 18/6, 721-723, Nov-Dec 1954

Abstract : Various opinions are presented regarding the H-bond in relation to the structure of aromatics. It is shown that the intramolecular H-bond, which creates an additional cycle with the participation of the hydrogen (the hydrogen cycle), affects the distribution of electron density in aromatic compounds. The intramolecular H-bond was found to stabilize the H-atom, it is not a deterrent for chem. reactions because of the H-atom but such

Institution: The A. I. Gertsen State Pedagogical Institute, Leningrad

Submitted :

Card 2/2 Pub. 43 - 47/62

Periodical : Izv. AN SSSR. Ser. fiz. 18/6, 721-723, Nov-Dec 1954

Abstract : reactions are usually very difficult because of the necessity of overcoming the H-bond effect. The problem of the H-atom in relation to H-bonds is discussed. Eight references: 4 USSR, 1 Swiss, 1 USA and 2 German (1891-1951). Table.

PEREKALIN, V. V.

USSR/ Chemistry - Reaction processes

Card 1/1 : Pub. 151 - 23/37

Authors : Perekalin, V. V., and Sorova, A. S.

Title : Reaction of nitrostyrene with certain compounds containing methylene groups activated by carbonyl and carboxyl groups

Periodical : Zhur. ob. khim. 24/3, 513-516, Mar 1953.

Abstract : The reaction of omega-nitrostyrene, with substances having mobile hydrogen atoms in methylene and methyl groups, was investigated. The hydrogen atoms were activated by electrophilic (carboxyl, carbonyl) groups oriented in alpha-position or in conjugation with the methylene or methyl groups. The effect of basic catalysts on the reaction of nitrostyrene with various compounds possessing methylene groups activated by carbonyl and carboxyl groups is explained. A new method for the synthesis of gamma-amino acid derivatives was introduced. Eight references: 5-USA; 2-German and 1-USSR (1879-1949).

Institution : The A. I. Gertsen Pedagogical Institute, Leningrad

Submitted : October 23, 1953

PEREKALIN, V. V.

A & dyes from 5-amino-1-naphthol and some of its derivative IX. The hydrogen bond in some *peri*-hydroxyazo dyes. V. V. Perekalin and Z. S. Segalica (A. I. Gertsen State Institute, Leningrad). *Zhur. Obshchei Khim.* 24, 653-7 (1954); cf. *C.A.* 45, 10049g. In some *peri*-hydroxyazo dyes, specifically, 8-benzeneazo-1-naphthol, the H atom of HO group is devoid of mobility owing to the formation of the intramolecular hydrogen bond. To 1.50 g. 5-amino-1-naphthol in 1 ml. 37% HCl and 200 ml. H₂O was added in 2 hrs. at 10° 100 ml. 0.1N PhN₂Cl and the mixture was stirred 5 hrs. and allowed to stand overnight. The pptd. dye was triturated with 10% NaOH, dild. to 100 ml., heated to 60° 8-10 min., cooled, filtered, and the filtrate acidified with AcOH yielding the alkali-sol. 8-benzeneazo-5-amino-1-naphthol. The ppt. was washed with H₂O and again treated with NaOH. There was obtained after crystallization from EtOH, 4-benzeneazo-5-amino-1-naphthol (I, m. 164-5°, brown, insol. in dil. NaOH, yielding blue solns. in H₂SO₄ which turned violet; the yield of the latter was 16%. This (1 g.) in 90% EtOH was treated hot with 1.5 g. Na₂S₂O₄ in weakly basic aq. soln, yielding a light yellow soln.; after filtration, the 4-amino-1-naphthol was not isolated but was directly oxidized by treatment at 6-7° with 10% aq. FeCl₃ which gave a red soln. which

was extd. with CHCl₃, the ext. was evapd. and the residue sublimed yielding brown II, m. 144-7°, which



in 10% NaOH gives a red soln. turning brown; in H₂SO₄ forms a red soln. To hot soln. of 1 g. I in 95% EtOH was added 3 ml. 66% H₂SO₄, followed by equimolar amount of fstd. NaNO₂ soln. (brown soln.); after removal of 15-20 ml. EtOH and cooling, the soln. was quenched in cold H₂O yielding brown 8-benzeneazo-1-naphthol m. 140-2° (from EtOH), insol. in cold or hot NaOH. Color of wood dyed with this after, or without, mordanting was the same.

G. M. Kuznetsov.

PEREKALIN, V. V.

USSR/Chemistry Reaction processes

Card : 1/1 Pub. 151 - 27/35

Authors : Perekalin, V. V., and Popova, L. P.

Title : Destruction of carbon-carbon bonds under the effect of diazo-compounds.
Part 1.- Reaction of some ternary amines of the triphenylmethane series
with diazo-compounds

Periodical : Zhur. ob. khim. 24, Ed. 7, 1226 - 1232, July 1954

Abstract : The reaction of ternary triphenylmethane amines with diazo-compounds
and the splitting of carbon-carbon bonds between the methane carbon atoms
were investigated. The attachment of one of the cleavage products
(dimethylaniline radical) to the diazo-compound, with consequent formation
of an azo-dye and the separation of the second product (benzaldehyde,
4-methylaminobenzaldehyde, formic acid) in free form, were determined
analytically. The effect of the amine structure and activity of the
diazo-compound, on the bond cleavage, is explained. Fourteen German,
4 USSR and 2 USA references. Tables.

Institution : The Gertsen Pedagogical Institute, Leningrad

Submitted : September 29, 1953

PEREKALIN, V. V.

USSR/Chemistry

Reaction processes

Card

: 1/1 Pub. 151 - 28/35

Authors

: Perekalin, V. V., Popova, L. P., and Abramovich, T. I.

Title

: Destruction of carbon-carbon bonds under the effect of diazo-compounds. Part 2.- Reaction of some ternary amines of the diphenylmethane series with diazo-compounds

Periodical

: Zhur. ob. khim. 24, Ed. 7, 1233 - 1238, July 1954

Abstract

: The cause for the splitting of the carbon-carbon bonds between the methane carbon atom and the carbon atoms of benzene nuclei, which takes place during the reaction of ternary diphenylmethane amines with diazo-compounds, is elucidated. The effect of the H-atom displacement by the hydroxyl group in the methane radical, and conversion of the carbinol radical to a carbonyl radical on the splitting of the carbon-carbon bonds, is explained. Three USSR, 4 German, and 1 Italian reference. Table.

Institution : The Gertsen Pedagogical Institute, Leningrad

Submitted : September 29, 1953

Reactions of diketene. II. Reaction of diketene with indole and its oxygen-containing derivatives. V. V. ~~Slavacheva~~ (A. I. Gerasimovskiy and N. I. Slavacheva) *Zh. Khim. Fiz.*, 28, 2184-5 (1955), *Chem. Abstr.* 47, 11155c. 1000. over 1 hr. of 3.36 g. diketene to 4.68 g. indole, 20 ml. C₆H₆, 20 ml. MePh and 0.5 ml. pyridine at -10°, heating 1 hr. on a steam bath, evapn. and treatment with much 2% NaOH, gave on neutralization of the acid with dil. HCl and CO₂ 43.7% *N*-acetoacetylindole (I), m. 100°. The yield is almost the same when quinoline or Me₂NPh is used as catalyst; with B₆N the yield is zero, however. Hydrolysis of the product in 2% NaOH gave indole and Me₂CO. Coupling with PhN₂Cl in aq. alc. NaOAc gave 43% *N*-acetoacetylphenylazindole, m. 178-80°, yellow, which hydrolyzed in 5% NaOH to *N*-phenylaracetate, m. 195°, in 91% yield. I refluxed with Ac₂O at 180-70° 3 hrs. gave 12.5% *N*-acetoacetyl-3-acetylindole, m. 178-9°, which hydrolyzed

with 5% NaOH to 3-acetylindole, m. 190-1°. Treatment of 3.36 g. diketene with 3.50 g. indoxylcarboxylic acid in C₆H₆ in the presence of 3 drops pyridine (45 min. on steam bath in H₂) gave 47% *N*-acetoacetylindoxyl, m. 110°. With a H₂ atm. the yield is 30%; hydrolysis with 5% HCl gave indigo and Me₂CO. Diketene reacts with phthalimide in MePh in the presence of pyridine at reflux (1.5 hrs.) yielding 07% *N*-acetoacetylphthalimidine, m. 135-7°, which hydrolyzed with aq. Na₂CO₃ to phthalimidine and Me₂CO. Diketene and dioxindole similarly gave 25% 2,3-diacetoacetylindole, m. 170-2°. Indole and phthalimide as well as oxindole failed to react owing to their relatively high acidity. G. M. Kosolapov

USSR.

✓ Synthesis of derivatives of γ -amino acids. V. V. Pere-
kalin and A. S. Sopova. Doklady Akad. Nauk S.S.S.R.
1954, 164, 1954. Condensation of $\text{PhCH}_2\text{CHNO}_2$ with
 $\text{CH}_3(\text{CO}_2\text{Et})_2$ in dry MeOH 2 hrs. at $30-5^\circ$ gave 61.4%
 $\text{PhCH}(\text{CH}_2\text{NO}_2)\text{CH}(\text{CO}_2\text{Et})_2$; m. 84° . This (5 g.) in MeOH
was added to 5 g. Ni catalyst (Adkins and Billica, C.A. 42,
3328c) in MeOH presatd. with H at room temp. and hydro-
genation continued 5 hrs. at $30-40^\circ$ gave 97% 3-phenyl-
4-hydroxy-3-pyrrolidine, m. 132° (from MeOH). This
(3 g.) refluxed 10 hrs. with 300 ml. 18% HCl gave 75.6%
 $\text{H}_2\text{NC}_4\text{H}_7\text{CHPhCH}_2\text{CO}_2\text{H}$, m. 209° (from dil. EtOH);
boiled in Ac_2O 3 hrs. this gave the N-1c analog, 70.4%
m. 65° (from hexane). G. M. Kosolapov

PEREKALIN, V. V.

AID P - 3171

Subject : USSR/Chemistry

Card 1/1 Pub. 119 - 6/8

Authors : Perekalin, V. V. and A. S. Sopova (Leningrad)

Title : Nitroolefins

Periodical : Usp. khim., 24, 5, 613-634, 1955

Abstract : The preparation of nitroolefins by nitration of aliphatic olefins and by condensation of aldehydes with primary saturated nitro compounds is reviewed. The physical and chemical properties of nitroolefins are discussed in some detail. Four tables, 158 references, 7 Russian (1873-1952).

Institution : None

Submitted : No date

PEREKALIN, V. V.

Reactions of diketene. III. Reaction of diketene with some aromatic hydroxy compounds. Synthesis of phenyl acetates. K. B. Rall and V. V. Perekalin (A. I. Gertsen Pedagog. Inst., Leningrad). *Dokl. Akad. Nauk SSSR*, 25, 278-81; *J. Gen. Chem. (U.S.S.R.)* 25, 269-64 (1955) (Engl. translation); cf. *C.A.* 30, 3908. To 4.7 g. PhOH in 30 ml. C_6H_6 was added a little pyridine followed, at -10° , by dropwise addn. of 1.5 g. diketene (I) over 10 min.; after refluxing 1.5 hrs. the mixt. yielded 72.3% $A: C_6H_5CO_2Ph$ (II), m. 50° ; a 63.9% yield was obtained similarly in Et_2O in the presence of few drops of H_2SO_4 . II (1.78 g.) in 100 ml. $EtOH$ and 10 ml. 0.1N $AcONa$ was treated with cooling over 2 hrs. with 110 ml. 0.1N $PhNH_2Cl$, yielding 64% $Ph_2CCHAc: N: NPh$, yellow, m. $45-6^\circ$; this refluxed with 5% HCl gave 80% $AcCH(CO_2H): N: NPh$, m. 153° . II in concd. H_2SO_4 gave after 20 hrs. at room temp. a low yield of 4-methylcoumarin, m. 80° . $p-MeOC_6H_4OH$ (6.2 g.) in C_6H_6 , treated with a little pyridine, followed by 4.5 g. I gave after 1.5 hrs. refluxing 68% $p-MeOC_6H_4O_2CCH_2Ac$, m. $92-3^\circ$ (with H_2SO_4 as catalyst the yield was 68.6%); this with $PhNH_2Cl$, as above, gave 83% $p-MeOC_6H_4O_2CCH(N: NPh)Ac$, yellow, m. $95-7^\circ$. Hydroquinone (3.3 g.) and 2.8 g. I, in C_6H_6 , in the presence of pyridine, gave after 2 hrs. at reflux and extn. with hot $CHCl_3$ a 91% yield of $p-HOC_6H_4O_2CCH_2Ac$, m. 102° , which is readily hydrolyzed with hot 5% $NaOH$. The use of an excess of I gave $p-C_6H_4(O_2CCH_2Ac)_2$ (III), m. $121-2^\circ$; the same formed from I and the mono deriv. in 84% yield. III and $PhNH_2Cl$, as above, gave 97% $p-C_6H_4(O_2CCH_2Ac)_2: N: NPh$, orange, m. $226-7^\circ$ (from Ac_2O). $p-MeC_6H_4OH$ and I in C_6H_6 with a little pyridine similarly gave 92.5% $p-MeC_6H_4O_2CCH_2Ac$, m. 68° . Similarly was obtained 74.5% $p-BrC_6H_4O_2CCH_2Ac$, m. 54° . I (0.14 g.) added over 1.5 hrs. to 1.2 g. salicylaldehyde and 0.1 g. dry $KOAc$ at $35-40^\circ$, then treated with 5% HCl , gave 83.7% 3-methylcoumarin, m. $120-1^\circ$. G. M. Keselapov

Perekalin, V. V.

USSR/ Chemistry - Synthesis methods

Card 1/1 Pub. 22 - 28/60

Authors : Rall', D. B., and Perekalin, V. V.

Title : New method of synthesizing coumarin derivatives

Periodical : Dok. AN SSSR 100/4, 715-717, Feb 1, 1955

Abstract : A hitherto unknown reaction between diketene and certain aromatic hydroxy compounds was investigated for the purpose of obtaining coumarin derivatives. The results obtained are described. Two references: 1 USA and 1 USSR (1939 and 1952).

Institution : The A. I. Gertsen State Pedagogical Institute, Leningrad

Presented by : Academician I. N. Nazarov, August 8, 1954

PEREKALIN, V.V.

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Ketene diester (diketene), V. V. Perekalin and T. A. ~~Uspenskaya~~. *Uspekhi Khim.* 25, 1481-1484 (1956). A detailed review, with 102 references through 1955, of prepolymerization, properties, and reactions of ketene diesters. G. M. I.

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PEREKALIN, V V

USSR/Organic Chemistry. Synthetic Organic Chemistry. E-2

Abs Jour : Ref Zhur = Khimiya, No. 8, 1957, 26710.

Author : Perekalin, V.V.; Lerner, O.M.

Inst :
Title : Interaction of Diketene with Amides of Carboxylic Acids.

Orig Pub : Zh. prikl, khimii, 1956, 29, No. 10, 1609.

Abstract : N-acetoacetylammides of acetic, n-butyric, n-caproic, phenylacetic, dipehnylacetic, benzoic, n-toluic and cinnamic acids were prepared by the interaction of diketene (I) with amides of carboxylic acids in presence of basic catalysts. Their structure was established by the reaction of azofication, by producing phenylhydrazones and by a counter-synthesis of chloroanhydrides of acids and

Card 1/2

PEREKALIN, V.V.

PEREKALIN, V.V.; LERNER, O.M.

Reaction of dinitrodiolefins with substances containing active
hydrogen atoms in methylene groups. Zhur.prikl.khim. 29 no.10:
1610-1611 0 '56. (MIRA 10:10)
(Olefins) (Chemical reactions)

PEREKALLA, V. V.
 9
 4E4g
 Chemistry of nitrobenzene. II. Reaction of nitrobenzene with substances containing active methyl groups. V. V. Perekalla and A. S. Polyanskaya. Zhur. Obshch. Khim., 27, 1933-4 (1957) cf. J. A. 40, 9632b. Refluxing 0.61 g. *ms*-methylacridine and 0.40 g. 2-nitrostyrene (I) in C_6H_6 5 hrs. gave 62% 1-(9-acridinyl)-2-phenyl-3-nitropropane (IA), m. 164.5° ($ClCH_2CH_2Cl$). Refluxing 1.40 g. I to 60 ml. C_6H_6 with 2.65 g. quinaldine methiodide and 3 drops Et_3N 8 hrs. gave 66% 1-(2-*N*-methylquinolyl)-2-phenyl-3-nitropropane iodide, m. 174.5° (MeOH). Similarly, methylbenzothiazole methiodide gave 62% 1-(2-*N*-methylthiazolyl)-2-phenyl-3-nitropropane iodide, m. 171.5° (MeOH). Addn. of 2 drops Et_3N to 3.80 g. *ms*-methylacridine and 3.87 g. "nitroisohexylene" (II) (cf. Bouveault and Wahl, Bull. soc. chim. France 29, 643 (1903)) in EtOH gave after 24 hrs. 2.4 g. ppt. of the 3 form of 1-(9-acridinyl)-2-isobutyl-3-nitropropane, m. 149° (EtOH), while concn. of the filtrate yielded 1.05 g. α -isomer, m. 105° (MeCO). II. Quinaldine and Et_3N in dry MeOH yielded 62% 1-(2-methylthiazolyl)-2-isobutyl-3-nitropropane iodide, m. 178°. II and methylbenzothiazole methiodide gave 67% 1-(2-*N*-methylthiazolyl)-2-isobutyl-3-nitropropane iodide, m. 183°. Refluxing 2.78 g. 2-furylnitroethene (III) and 3.80 g. *ms*-methylacridine in C_6H_6 with 2 drops Et_3N 5 hrs. gave 66% 1-(9-acridinyl)-2-furyl-3-nitropropane, yellowish, m. 168° (C_6H_6); the yield was 65% if the mixt. was kept overnight without the catalyst. III and quinaldine methiodide in

Perekalyn, V. V.; Polyanitskaya, A. S.

C_6H_5 with a little BH_3 refluxed 1 hr. gave 70% 1-(2-N-methylaminolyl)-2-furyl-3-nitropropane iodide, brown, m. 161° (EtOH); at room temp. 24 hrs., the yield was 84%. Methylbenzothiazole methiodide and III similarly gave 60% brown 1-(2-N-methylthiazolyl)-2-furyl-3-nitropropane iodide, m. 183.5° (MeOH). Hydrogenation of IA over Raney Ni in MeOH gave 45% 1-(dihydro-9-acridinyl)-2-phenyl-3-aminopropane, isolated as HCl salt, m. 238°. Refluxing IA with 18% HCl 6 hrs. gave 80% 3-(9-acridinyl)-2-phenylpropanoic acid, m. 255°. No salt, silky needles, showing blue fluorescence in ultraviolet light. G. M. Kozlovskii.

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dim. v. len

PEREKALIN, V.V.; PADVA, G.D.

Reactions of diketene. Part 5: Reaction of diketene with dioxyraphthaline. Zhur. ob. khim. 27 no.9:2578-2585 S '57. (MIRA 11:3)

1. Leningradskiy pedagogicheskiy institut imeni A.I. Gertsena.
(Ketene) (Naphthaline)

PEREKALIN, V. V.

Dieter: 443/4E33/4E29(1)

Synthesis of derivatives of β -diketones. V. V. Perekalin and K. S. Parfenova. Zhur. Prikl. Khim. 36, 1270-1271 (1957); cf. C.A. 49, 8180r. A mixt. of 0.140 g. dimedon (I) and 0.149 g. nitrostyrene in 5 ml. abs. MeOH gave 65% of a white cryst. product, m. 153° (MeOH) (decompu.). 1 (0.54 g.) and 0.5 g. nitroisobutane in 10 ml. abs. MeOH, a week at room temp., gave a cryst. product, m. 152-3° (decompu.). Indandione (0.146 g.) and 0.149 g. nitrostyrene in 5 ml. CCl₄ contg. a drop of H₂N after 1 hr. at room temp. gave an oil which crystd. on stirring with a few drops of MeOH, m. 195° (MeOH-C₆H₆), yield 40%. To a mixt. of 0.444 g. phenylindandione, 0.288 g. nitrostyrene, and 5 ml. CCl₄, a drop of Et₃N was added. The oily product after 1 hr. at room temp. and evapn. of the solvent, m. 184-5° (Me₂CO), yield 53%. J. Benard

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PURCELLIN, V.V.; POLYANSKAYA, A.S.

Interaction between nitro olefines and compounds with active
methyl groups. Dokl. AN SSSR 112 no.3:441-444 Ja '57. (MLRA 10:4)

1. Predstavleno akademikom I.N. Nazarovym.
(Olefins) (Methyl group)

AUTHORS: Zobocheva, M. M., Perekalin, V. V. SOV/156-58-4-32/49

TITLE: The Interaction of Nitro-Olefins With Dimethyl Malonic Ester
(Vzaimodeystviye nitroolefinov s malonovodimetilovym efirom)

PERIODICAL: Nauchnyy doklady vysshey shkoly. Khimiya i khimicheskaya
tekhnologiya, 1958, Nr 4, pp 740-742 (USSR)

ABSTRACT: A detailed investigation of the interaction of the nitro-olefins
with dimethyl malonic ester was carried out. The reaction
process is as follows:

$$\begin{array}{c} \text{O}_2\text{NCH}=\text{CH} \\ | \\ \text{R} \end{array} + \text{CH}_2(\text{COOCH}_3)_2 \longrightarrow \begin{array}{c} \text{O}_2\text{NCH}_2-\text{CH}-\text{CH}(\text{COOCH}_3)_2 \\ | \\ \text{R} \end{array}$$
 The equimolar quantity of nitro-olefin in dry methanol solution
is added to the solution of dimethyl malonic ester in dry
alcohol at a temperature below 20°C and by intensive inter-
mixing. After 30 minutes of intermixing of the components, the
reaction mixture is acidified by acetic acid under addition of
finely disperse ice. The eliminated condensation products are
filtered off or extracted by ether, if liquid products are
formed. The products are purified by crystallization or distil-
lation in vacuum. The synthesized products were chemically

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SOV/156-58-4-32/49

The Interaction of Nitro-Olefins With Dimethyl Malonic Ester

analyzed and the values are given in table 2.
There are 2 tables and 5 Soviet references.

ASSOCIATION: Kafedra organicheskoy khimii Leningradskogo gosudarstvennogo pedagogicheskogo instituta im. A. I. Gertsena (Chair of Organic Chemistry at the Leningrad State Pedagogic Institute imeni A. I. Gertsen)

SUBMITTED: June 16, 1958

Card 2/2

AUTHORS: Perekalin, V. V., Sopova, A. S.

79-28 3-24/6

TITLE: The Synthesis of γ -Aminoketones
(Sintez γ -aminoketonov)

PERIODICAL: Zhurnal Obshchey Khimii 1958, Vol 28, Nr 3 pp. 675-679
(USSR)

ABSTRACT: The effectively realized reaction of nitroolefines with acetic ester successfully realized by the authors offered the possibility to make use of this reaction for the elaboration of a new synthesis of γ -aminoketones. These ketones have hitherto not been investigated as there seemed to be no possible ways for their synthesis as well as no possibilities of heterocyclization forming multiple pyrrolidine derivatives of interest for chemists; it was of interest to find the dependence on their structure of the capability of condensation of unsaturated nitrocompounds with acetic ester. The reaction of nitrostyrene with acetic ester on a catalytic contact with triethylamine and pyridine led to the formation of the ethylester of the 2-acetyl - 3 - phenyl - 4 - nitrobutyric acid (I) (98-82 %). The catalytic reduction of this ester

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The Synthesis of β -Aminoketones

75-28 3-24/61

resulted in two products, depending on the reaction period: the hydration for 10 hours only touched the nitrogroup and led to the formation of ethylesters of the 2-acetyl-3-phenyl-4-aminobutyric acid (II), that for 48 hours however, was accompanied by the saponification of the ester and the formation of 2-acetyl-3-phenyl-4-aminobutyric acid (III). The ester of the aminoketonic acid (II) was subjected to different chemical reactions: 1. The saturation of a benzene solution with dry hydrochlorine furnished the hydrochloric salt. ((IV) 2. The action of acetylchloride gave the acetyl derivative (V). 3. The heating with a 10 % hydrochloric acid led to the solution of the basic problem: As a consequence of the ketone cleavage the β -aminoketone-1-amino-2-phenylpentanon-4 (IV) in form of hydrochloric salt resulted with a yield of 45 %. The aminoacid, in heating above the melting point, easily converted to the pyrrolidine derivative by heterocyclization; this again was converted to the hydrochloric salt of 3-phenyl-4-acetylpyrrolidin-5 (VII) by dry hydrochlorine. Special attention was paid to the structure of the condensation product (I) which was to be determined. It formed in an alcohol medium with phenylhydrazine in the presence of hydrochlorine acid the phenyl-

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The Synthesis of γ -Aminoketone

79-28-3-24/61

methylpyrazolone derivative (VIII), which is identical to the reaction product of nitrostyrene with phenylmethylpyrazolone; hydrazone (IX) synthesized from (I) was converted to pyrazolone (VIII) by heating in benzene in the presence of phosphorus pentoxide. Thus the structure of the condensation product of nitrostyrene with acetic ester is clearly proved.

There are 3 references; which are Soviet

ASSOCIATION: Leningradskiy pedagogicheskiy institut imeni Gertsena
(Leningrad Pedagogical Institute imeni Gertsen)

SUBMITTED: January 21, 1957

Card 3/3

AUTHORS: Perekalin, V. V., Lerner, O. M.

SOV/79-18-7-19/64

TITLE: The Conversion of Dinitro-Olefines With Compounds Having Mobile Hydrogen Atoms in the Methyl Groups (Vzaimodeystviye dinitroolefinov s soyedineniyami, soderzhashchimi podvizhnyye vodorodnyye atomy v metilenovykh gruppakh)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol 28, Nr 7, pp 1815 - 1823 (USSR)

ABSTRACT: In completion of earlier papers and articles written by the same authors (Refs 1-8) it seemed useful to them to improve certain methods of the synthesis of some dinitro-olefines, viz. derivatives of terephthalaldehyde, and to investigate their conversion with compounds having mobile hydrogen atoms in the methyl groups. When using ethylene diamine (Ref 9) as catalyst the authors modified known methods of the condensation of terephthalaldehyde with nitromethane (Refs 5,10) and in some cases obtained a considerably higher yield of dinitro-olefines (Table). The synthesized dinitro-olefines were caused to react with a great number of compounds having mobile hydrogen atoms in their methyl groups, viz. with malonic ester and acetic ester,

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The Conversion of Dinitro-Olefines With Compounds
Having Mobile Hydrogen Atoms in the Methyl Groups

SOV/19-26-7-19/64

with phenyl-methyl pyrazolone, cyanacetic- and nitroacetic esters, as well as with phenylnitromethane. The nitro groups at various molecule terminals in the above mentioned nitro-olefines produce two combined systems in an opposite direction, which fact was of interest for the reactivity of such compounds, and which has hitherto not been investigated. It was found that the presence of two combined systems in an opposite direction in the dinitro-olefines is no hindrance for the condensation course with active methylene components under the action of two nitro-vinyl groups. There are 1 table and 14 references, 5 of which are Soviet.

ASSOCIATION: Leningradskiy pedagogicheskiy institut imeni A.I.Gertsena
(Leningrad Pedagogical Institute imeni A.I.Gertsen)

SUBMITTED: June 17, 1957

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The Conversion of Dinitro-Olefines With Compounds
Having Mobile Hydrogen Atoms in the Methyl Groups

SOV/7-28-7-19/64

1. Dinitroethylenes--Chemical reactions
2. Methyl radicals--Chemical reactions
3. Condensation reactions

Card 3/3

SOV/20-121-2-28/53

AUTHORS: Abramovich, T. I., Gragerov, I. P., Perekalin, V. V.

TITLE: The Isotopic Exchange of Hydrogen and the Capability of the Methyl Derivatives of Nitrogen Containing Heterocycles to Enter Into Condensation Reactions (Izotopnyy obmen vodoroda i sposobnost' metil'nykh proizvodnykh azotsoderzhashchikh geterotsiklov k reaktsiyam kondensatsii)

PERIODICAL: Doklady Akademii nauk SSSR, 1958, Vol. 121, Nr 2, pp. 295 - 298 (USSR)

ABSTRACT: The heterocycles mentioned in the title which have a methyl radical in an α - or β -position to the hetero atom, as well as their quaternary salts enter into condensation reactions with aldehydes, nitroso-compounds, diazo-compounds, nitroolefines (Ref 1) and others. These conversions usually take place in an alkaline medium. These compounds can be arranged into a series as regards their activity. The opinion (e.g. as mentioned in Ref 2) that the velocity of the condensation reactions as well as the possibility of their course at all is determined by the easy cleaving-off of protons from the

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SOV/20-121-2-28/53

The Isotopic Exchange of Hydrogen and the Capability of the Methyl Derivatives of Nitrogen Containing Heterocycles to Enter Into Condensation Reactions

methyl radicals, is widely spread. As the said easiness is quantitatively characterized by the velocity of the isotopic exchange of hydrogen in an alkaline medium it was interesting to compare the velocity of the exchange of the compounds mentioned with their tendency to condensation reactions. By doing so also the factors could be explained which determine the mobility of the hydrogen in the methyl group in dependence on its structure. In the present paper the authors investigated the exchange of hydrogen between the compounds of the series (1) and the methylalcohols CH_3OD in the presence of triethylamine. The ratio between the substance investigated, the alcohol, and the catalyst was about constant and amounted to 0,007 : 0,03 : 0,001 moles correspondingly. Figure 1 presents the results obtained as the function of $\ln(1 - z)$ versus the duration t , where z denotes the share in the exchange calculated in relation to the three hydrogen atoms of the methyl radical subjected to the exchange. The points obtained in the experiment may satisfactorily be located on the straight line describing the time course of the reactions of first order. The mean

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SOV/20--21-2-28/53

The Isotopic Exchange of Hydrogen and the Capability of the Methyl Derivatives of Nitrogen Containing Heterocycles to Enter Into Condensation Reactions

values of the velocity constants K (Table 1) were found from the slope of this straight. The exchange of the above mentioned experiments concerns only the hydrogen of the methyl residues being in an α - or β -position to the hetero atom (the hydrogen of the N-H bindings is not included). The consecutive order of the exchange velocities reflects the differently easy cleaving-off of protons from the methyl radicals. The comparison between the series (1) and the data in table 1 shows that the activity in the condensation reactions does not correspond to the tendency to the cleaving-off of protons. Thus the ionization of hydrogen, at least in several cases, does not represent the limiting stage of condensation reactions. There are 1 figure, 1 table, and 11 references, 8 of which are Soviet.

ASSOCIATION: Leningradskiy pedagogicheskiy institut im. A. I. Gertsena
(Leningrad Pedagogical Institute imeni A. I. Gertsen)
Institut fizicheskoy khimii im. L. V. Pisarzhevskogo Akademii
nauk USSR (Institute of Physical Chemistry imeni L. V.

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SOV/20-121-2-28/53

The Isotopic Exchange of Hydrogen and the Capability of the Methyl Derivatives
of Nitrogen Containing Heterocycles to Enter Into Condensation Reactions

Pisarzhevskiy, AS UkrSSR)

PRESENTED: March 18, 1958, by A. N. Frumkin, Member, Academy of Sciences,
USSR

SUBMITTED: March 17, 1958

Card 4/4

AUTHORS: Bobovich, Ya.S., Perekalin, V.V. SOV/20-121-6-21/45

TITLE: An Investigation of the Structure of Unsaturated Nitrocompounds by Means of the Raman Effect (Issledovaniye stroyeniya nepredel'nykh nitrosoyedineniy metodom kombinatsionnogo rasseyaniya sveta)

PERIODICAL: Doklady Akademii nauk SSSR, 1958, Vol 121, Nr 6, pp 1028 - 1030 (USSR)

ABSTRACT: A very important factor for a successful reciprocal reaction of the above mentioned compounds with nucleophilic reagents (in particular with compounds containing mobile hydrogen atoms in the methyl- and methylene groups) is the electron deficiency ($+\delta$) on the α -carbon atom of the nitroolefin-ethylene-radical. The magnitude of this deficiency which is due to the conjugation of the nitro group with the double bond, depends to a considerable degree on the chemical structure of these nitrocompounds (on the nature of the radicals bound to the ethylene radical). For the solution of various synthetic problems, sufficient objective knowledge of the character of the conjugation in the nitroolefines was required. For this purpose this investigation of the bands of intensity of 22 unsaturated nitroderivatives, further of nitromethan and some aromatic nitroderivatives was performed.

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An Investigation of the Structure of Unsaturated
Nitrocompounds by Means of the Raman Effect

SOV/20-121-6-21/45

The spectra of most of the compounds were determined for the first time. As already known (Ref 1), the intensity values of the spectra (lines) in question change according to conjugation and therefore make it possible to estimate the latter. Besides, in this way the judgement of local changes of the electron cloud instead of the behaviour of the molecule taken in its totality is rendered possible. The results are summarized in table 1. The investigation proved that the intensity of the lines is widely variable (Table 2). For the same molecules the intensity of the antisymmetrical oscillation of the benzene ring is liable to considerable changes. The authors conclude as follows:

- 1) The unsaturated olefines represent a uniform, conjugated system.
- 2) In para-dinitroolefines (XVIII and XIX) the conjugation increases rapidly, as compared both with the corresponding meta-isomers (XXI and XXII) and with mono-nitroolefines (VI and VII).
- 3) The methyl groups on the ethylene carbon, which is combined with the nitro group, diminish the conjugation.

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- 4) The degree of depolarization of the symmetric oscillation (def 2) ranges for most of the compounds between 0,22 and 0,43. A.N. Terenin, Member, Academy of Sciences, USSR, and Professor B.S. Neporent have participated in this study and have made possible the spectral measuring.

There are 2 tables and 2 references, which are Soviet.

PRESENTED: April 14, 1958, by A.N. Terenin, Member, Academy of Sciences, USSR.

SUBMITTED: April 2, 1958

Card 3/3

5(3)
AUTHOR: Perekalin, V. V. SOV/79-29-9-20/76
Zobacheva, M. M.
TITLE: Synthesis of γ -Amino Acids and Pyrrolidones
PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 9, pp 2905-2910 (USSR)
ABSTRACT: A survey of the synthesis of γ -amino acids and pyrrolidones described in publications reveals that their application is often restricted by the difficult preparation of the initial products. Known methods fail to produce more complicated γ -amino acids. In the investigation under review, the sodium derivative of dimethyl ester of malonic acid was caused to react with nitro-olefins of the aliphatic, aromatic, heterocyclic series, i.e. with β -isopropyl nitroethylene (Ref 10), p-methoxy-O-nitrostyrene (Ref 11), p-oxy-m-methoxy-O-nitrostyrene (Ref 12), β -furyl nitroethylene (Ref 13), β -thienyl nitroethylene (Ref 14); this condensation resulted in compounds (I) which, by reduction over a skeleton nickel catalyst, yielded pyrrolidones (II); the acid hydrolysis of carbomethoxy pyrrolidones led to substituted γ -amino butyric acids (III), and the alkaline hydrolysis to pyrrolidone carboxylic acids (IV). On heating, acids (III) and (IV) transformed into pyrrolidones (V); the hydrolysis of pyrrolidones again yielded γ -amino acids. A synthesis was also made of the acetyl compounds (VI) (Scheme).
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Synthesis of γ -Amino Acids and Pyrrolidones SOV/79-29-9-20/76

To prevent nitro-olefin from polymerizing, nitro-olefin, likewise solved in methanol, was added to the solution of sodium methyl malonate in anhydrous methanol, and not the other way round. Condensation took place on cooling; a considerable resinification occurred above 20°. The nickel catalyst applied was first saturated with hydrogen; from the amount of absorbed hydrogen it was possible to deduct that only the nitro group had been reduced, and not the carboxyl groups. Acids (IV) were separated by diluted hydrochloric acid from the alkali lyes resulting after the hydrolysis of compounds (II). On heating to the melting point they were transformed into the corresponding pyrrolidones. Their hydrolysis with 10% caustic potash solution and subsequent neutralization with diluted hydrochloric acid, yielded the γ -amino acids as the end product. Five γ -amino acids (IIIa), (IIIb), (IIIv), (IIIg), (IIIh), and, correspondingly, five pyrrolidones (Va), (Vb), (Vv), (Vg), (Vd) were synthesized. Melting point, yields and data supplied by the elementary analysis of the products obtained are shown by the table. There are 1 table and 14 references, 4 of which are Soviet.

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Synthesis of γ -Amino Acids and Pyrrolidones SOV/79-29-9-20/76

ASSOCIATION: Leningradskiy pedagogicheskiy institut imeni A. I. Gertsena
(Leningrad Pedagogical Institute imeni A. I. Gertsen)

SUBMITTED: July 11, 1958

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5(3)

SOV/80-32-4-47/47

AUTHORS: Perekalin, V.V. and Lerner, O.M.

TITLE: The Condensation of Isophthalic Aldehyde With Nitromethane (Kondensatsiya izoftalevogo al'degida s nitrometanom)

PERIODICAL: Zhurnal prikladnoy khimii, 1959, Vol 32, Nr 4, pp 939-940 (USSR)

ABSTRACT: The condensation of isophthalic aldehyde with nitromethane was already studied by Ruggli and Schetty [Ref 6] who obtained dinitrodiolefin with a yield of 35% by using methylamine and benzylamine as catalysts. The authors of the present note continued to study this reaction and achieved an increase in the yield of dinitrodiolefin, which attained 67%. The method for increasing the yield of the final product was the use of the aqueous concentrated solution of caustic soda as a condensing means taken in excess. The experimental part of the investigation and

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SCV/80-32-4-47/47

The Condensation of Isophthalic Aldehyde With Nitromethane

the various phases of reactions are described in detail.
There are 9 references, 3 of which are Soviet, 3 German, 2 American
and 1 Swiss.

SUBMITTED: July 11, 1958

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USCOMM-DC-60,915

5(3)

SOV/80-32-5-51/52

AUTHORS: Kon'kova, V.A., Perekalin, V.V.

TITLE: The Interaction of Nitrocompounds With Barbituric Acid

PERIODICAL: Zhurnal prikladnoy khimii, 1959, Vol 32, Nr 5, pp 1178-1179 (USSR)

ABSTRACT: The products of barbituric acid containing nitro- and amino-groups in alkyl residues bound to the methylene atom of carbon are little investigated. In the article reactions of barbituric acid with various unsaturated nitro-compounds are studied and the following compounds are synthesized: 5,5-(2',2'-dinitrodiethyl)-barbituric acid; 5-[1-(nitromethyl)-butyl(1)]-barbituric acid; 5-[1-(1-nitromethyl-3-methyl)-butyl]-barbituric acid; 5,5-[(2'-nitroethyl-1-nitromethyl)-butyl]-barbituric acid.

SUBMITTED: There are 2 references, 1 of which is Soviet and 1 American.
July 10, 1958

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5(3)

SOV/20-124-3-23/67

AUTHORS: Perekalin, V. V., Parfenova, E. S.TITLE: The Synthesis of Derivatives of Cyclic β -Diketones (Sintez proizvodnykh tsiklicheskikh β -diketonov)

PERIODICAL: Doklady Akademii nauk SSSR, 1959, Vol 124, Nr 3, pp 592-594 (USSR)

ABSTRACT: The cyclic β -diketones (such as, e.g., phenyl indandione-1,3) are becoming increasingly important in pharmacology as anti-coagulants, spasmolytics, as well as for their effect which resembles that of atropine. - The condensation of various cyclic β -diketones (dimedon, indandione-1,3 and 2-phenyl-indandione-1,3 as well as 4-oxycoumarin) was carried out, at room temperature, with a series of aliphatic, aromatic and heterocyclic unsaturated nitro-compounds (nitroethylene, β -nitrostyrene, furyl-nitroethylene, and thienyl-nitroethylene), in the presence of basic catalysts (methyl sodium, triethylamine) in organic solvents (methanol, benzene). To dimedon, only nitroethylene affiliated with two molecules; the aromatic nitroolefins formed only 1:1 adducts. Indandione, on the other hand, reacted with all of the above-mentioned

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SOV/20-124-3-28/67

The Synthesis of Derivatives of Cyclic β -Diketones

nitroolefins with both hydrogen atoms of the methylene group. 4-Oxycoumarin could be only condensed with β -nitrostyrene. The aliphatic nitroolefins polymerized. The aromatic substituted nitroolefins which possess nucleophile substituents at the benzene ring (4-dimethylamino- and 4-methoxy groups) did not react with oxycoumarin, because of reduced activity. Highly acid diketones such as 2-nitrodimedon, 2-nitroindandione-1,3 and 2-indandione-1,3-sulfo acid, do not react with nitroolefins. The reaction medium exerts a strong influence on the course of the reaction. Non-polar solvents (benzene) prevent the enolization of the cyclic β -diketones, thus promoting the reaction, whereas the polar solvents (methanol) have the opposite effect, thus the yield is smaller. - The structure formulae and the physical data of the synthesized compounds are listed in tables. - The paper presents a general method for the synthesis of derivatives of cyclic β -diketones, characterized by the fact that the hydrogen atoms of their methylene groups are replaced by a nitroethyl group connected with various aliphatic, cyclic or heterocyclic radicals. There are 3 tables, and 2 references, 1 of which is Soviet.

Card 2/3

5(4)

AUTHORS: Bobovich, Ya. S., Perekalin, V. V. SOV/24-127-6-26/51

TITLE: Investigation of the Structure of Some Ethylene and Styrene Derivatives by Means of Raman Radiation

PERIODICAL: Doklady Akademii nauk SSSR, 1959, Vol 127, Nr 6, pp 1239-1241 (USSR)

ABSTRACT: The activity of unsaturated organic nitro-compounds is subject to the conjunction of the nitro group with the double bond. In order to determine the influence upon activity when replacing the nitro group by other electrophilic groups (nitrile-, carbonyl-, carboxylic-, sulfonic-, phosphoric-groups) in conjunction, the intensity of the Raman lines corresponding to the double bond was measured. The method was already described in reference 1. Table 1 shows the frequencies and intensities (relative to benzene double linkage = 1) for ethylene derivatives, table 2 for styrene derivatives. The introduction of the nitro group into an ethylene derivative multiplies the intensity. The intensity of vinylsulfonic and vinylphosphonic compounds is - contrarily as expected - lower than in ethylene derivatives with isolated double bond. The methylene group acts like a barrier if the nitro group is separated from the double bond

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Investigation of the Structure of Some Ethylene and
Styrene Derivatives by Means of Raman Radiation

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by the methylene group, and no increase of intensity occurs. For chloro- and bromo-allyl, however, as well as for halogen compounds of styrene no barrier effect, caused by the methylene group, may be observed. No interpretation may be given yet to explain this specific property of the halogens. In common the styrene compounds satisfy the same rules. There are 2 tables and 7 references, 5 of which are Soviet.

PRESENTED: April 23, 1959, by A. N. Terenin, Academician

SUBMITTED: April 2, 1959

Card 2/2

5.3610

68163

~~5(3)~~

AUTHORS:

Perekalin, V. V., Lerner, O. M.

SOV/20-129-6-29/69

TITLE:

Synthesis of Conjugated Dinitrodiene⁷

PERIODICAL:

Doklady Akademii nauk SSSR, 1959, Vol 129, Nr 6, pp 1303 - 1305
(USSR)

ABSTRACT:

Aliphatic unsaturated nitro compounds with 2 conjugated nitrovinyl groups had not been described in publications. Since the structure and reactivity of such dinitrodienes are of interest, the authors synthesized the simplest dinitrodiolefine: 1,4-dinitrobutadiene-1,3 (V). They started from 1,4-dinitrobutene-2 (I). Usable directions for the preparation of 1,4-dinitrobutene in the only way possible (by addition of nitrogen tetroxide to divinyl) are missing in the publications (Refs 2-5). It was found that a rapid addition of N_2O_4 to divinyl under vaccination with crystalline I is necessary for successful nitration. 1,4-dinitrobutene-2 was transformed into fumaric acid (II) by acid hydrolysis to prove the structure; thus, a simple method of synthesis of fumaric acid and, consequently, of maleic acid anhydride was found (Ref 6). Contrary to data from publications (Ref 5), 1,4-diaminobutene-2 (III) was obtained in the reduction of I with tin and hydrochloric acid, and not 1,4-diaminobutane.

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Synthesis of Conjugated Dinitrodiene

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In the presence of iodine traces, I slowly deposits chlorine and turns into 2,3-dichloro-1,4-dinitrobutane (IV). This compound is very unstable as compared with alkalis and organic bases. On account of the Raman spectra, the double bond in 1,4-dinitrobutene-2 (contrary to nitroallyl) participates in the conjugation with the nitro groups (Ref 7). HCl is separated during the effect of lead acetate in glacial acetic acid on IV, and 1,4-dinitrobutadiene-1,3 (V) is formed. This synthesis apparently is the first example of using lead acetate for the transformation of 1,2-halogen-nitroalkanes into unsaturated nitro compounds. V proved to be a very resistant compound. It did not enter the diene-synthesis reaction, did not react with the active hydrogen atoms of the methylene groups as do mononitroolefines, and could be slowly brominated. Thus, a dibromide was formed. The intensity of the bands of the double bond in the Raman spectrum of V (Table 1) exceeds the respective value of the mononitroolefines by about one order of magnitude. This indicates the presence of a conjugated system. There are 1 table and 8 references, 3 of which are Soviet.

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Synthesis of Conjugated Dinitrodiene

SOV/20-125-6-29/69

PRESENTED: July 11, 1959, by M. I. Kabachnikov, Academician

SUBMITTED: July 13, 1959

Card 3/3

BOBDEVICH, Ya.S.; ~~PEREVALIN~~, V.Y.

Investigation of the structure of some unsaturated compounds by means of Raman spectra. Zhur. strukt. khim. 1 no.3:313-318 S-O '60. (MIRA 14:1)

1. Gosudarstvennyy opticheskly institut imeni S.I. Vavilova, Leningrad. (Unsaturated compounds--Spectra)

5.3600

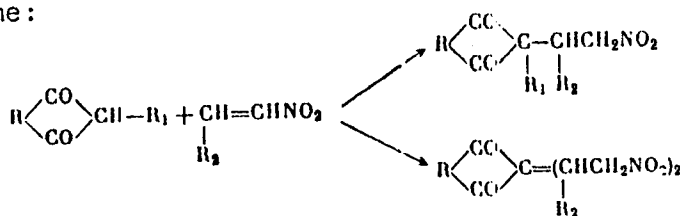
77857
SOV/79-30-2-2/13

AUTHORS: Perekalin, V. V., Parfenova, K. S.

TITLE: Chemistry of Unsaturated Nitro Compounds. VI. Synthesis of Derivatives of Cyclic β -Diketones

PERIODICAL: - Zhurnal obshchey khimii, 1960, Vol 30, Nr 2, pp 338-393 (USSR)

ABSTRACT: The authors have effected, for the first time, reactions of many cyclic β -diketones with a series of aliphatic, aromatic, and heterocyclic unsaturated nitro compounds. The reactions proceed by the following scheme:



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Chemistry of Unsaturated Nitro
Compounds. VI

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melting points are shown in Table 1.

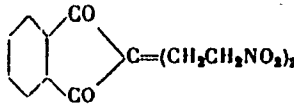
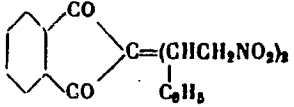
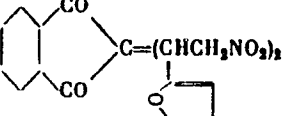
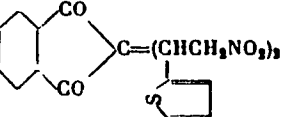
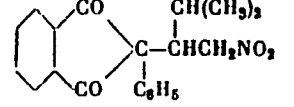
Table 1. (a) Numbers of the compounds; (b) formula;
(c) mp (solvent); (d) yield (%).

(a)	(b)	(c)	(d)
(I)	$(CH_3)_2C \begin{array}{l} \diagup CH_2-CO \\ \diagdown CH_2-CO \end{array} C \equiv (CH_2CH_2NO_2)_2$	$\begin{array}{l} 104.5^\circ \\ \text{(methanol)} \end{array}$	52
(II)	$(CH_3)_2C \begin{array}{l} \diagup CH_2-CO \\ \diagdown CH_2-CO \end{array} CH-CHCH_2NO_2$ $ $ C_6H_5	$\begin{array}{l} 137.2 \\ \text{(methanol)} \end{array}$	65
(III)	$(CH_3)_2C \begin{array}{l} \diagup CH_2-CO \\ \diagdown CH_2-CO \end{array} CH-CHCH_2NO_2$ $ $ C_6H_5OH, OCH_3, \dots	$\begin{array}{l} 174 \\ \text{(methanol)} \end{array}$	47

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(Table cont'd on Card 4/8)

77857 SOV/79-30-2-8/78

(IV)		111 (methanol)	35
(V)		188.5 (methanol- benzene)	85
(VI)		156.4° (methanol- benzene)	56
(VII)		212-213 (dioxan)	81
(VIII)		138.5 (methanol)	60

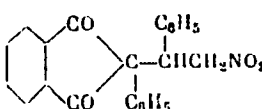
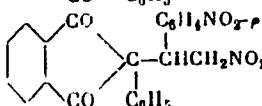
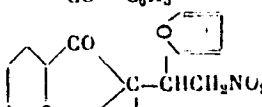
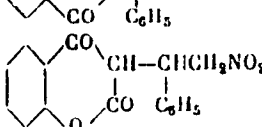
Card 4/8

(Table cont'd on Card 5/8)

Chemistry of Unsaturated Nitro
Compounds. VI

77857

SOV/79-30-2-8/78

(IX)		164.2 (methanol- benzene)	94
(X)		193 (methanol- benzene)	87
(XI)		132 (methanol- benzene)	55
(XII)		152 (ethanol)	50

The cyclic β -diketones having strong acidic properties (2-nitrodimedone, 2-nitroindandione-1,3 and 1,3-indandione-2-sulfonic acid) and ketones with very weak acidic properties (cyclohexanone, 1-indandione), as well as perinaphthoindandione,

Card 5/8

Chemistry of Unsaturated Nitro
Compounds. VI77857
SOV, 79-30-2-8/78

do not react with nitroolefines. (Thiophene was supplied by Professor V. Treibs (Treibs) (Leipzig); 2-nitrodimedone and 1,3-indandione-2-sulfonic acid were supplied by E. Yu. Gudriniyetse.) Thus, there exists an optimum acidity of the cyclic β -diketones, contingent for the reaction with nitroolefines. Hydrogenation of some of the nitroproducts led to formation of cyclic compounds, derivatives of pyrroline (see XIII in Table 2).

Table 2. (a) Numbers of the compounds; (b) formula; (c) mp (solvent); (d) yield (%).

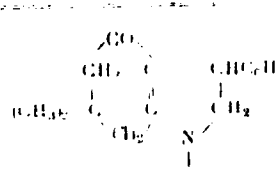
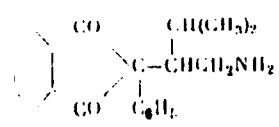
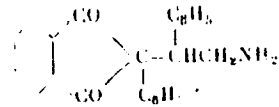
Card 6/8

Chemistry of Unsaturated Nitro
Compounds. VI

77857

SOV/79-30-2-8/78

Table 2.

(a)	(b)	(c)	(d)
(XIII)		237 ⁰ (methanol)	54
(XIV)	Hydrochloride of (XIII)	257 (methanol)	—
(XV)		168 (methanol)	43
(XVI)		216	33

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Chemistry of Unsaturated Nitro
Compounds. VI

77857
SOV/79-30-2-8/78

Compounds VIII and IX formed amines (XV and XVI).
There are 3 tables; and 20 references, 8 Soviet, 9
German, 1 U.K., 2 U.S. The 3 U.K. and U.S. refer-
ences are: G. Bucklay, P. W. Scaife, J. Chem. Soc.,
1472 (1947); W. King, F. Nord, J. Org. Ch., 14, 405
(1949); N. L. Drake, A. B. Ross, J. Org. Ch., 23,
717 (1958).

ASSOCIATION: Leningrad A. I. Gertsen Pedagogical Institute
(Leningradskiy pedagogicheskiy institut imeni A. I.
Gertsena)

SUBMITTED: February 2, 1959

Card 8/8

5.3610

78286

SOV/79-30-3-40/69

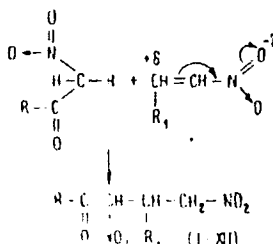
AUTHOR: Perekalin, V. V., Bayer, K.
TITLE: Synthesis of α , γ -Dinitroketones
PERIODICAL: Zhurnal obshchey khimii, 1960, Vol 30, Nr 3,
pp 943-945 (USSR)
ABSTRACT: Reaction of ω -nitroacetophenone, p-methyl-,
p-methoxy-, m, p-methylenedioxy- ω -nitroacetophenones
with β -nitrostyrene, p-methoxy-, p-nitro- β -nitro-
styrenes, 1,4-bis(β -nitrovinyl)benzene, 2-furyl- and
2-thienylnitroethylene yields several α , γ -dinitro-
ketones shown in the Table.



Card 1/5

Synthesis of α, γ -Dinitroketones

78286

SOV/79-30-3-40/69



- (I) $R = C_6H_5$, $R_2 = C_6H_5$; (II) $R = C_6H_5$, $R_2 = C_6H_4NO_2$; (III) $R = C_6H_5$, $R_2 = C_6H_4OCH_2CH_2$; (IV) $R = C_6H_5$, $R_2 =$ ; (V) $R = C_6H_5$, $R_2 =$ ; (VI) $R = C_6H_4OCH_2CH_2$, $R_2 = C_6H_5$; (VII) $R = C_6H_4CCH_2CH_2$, $R_2 = C_6H_4NO_2$; (VIII) $R = C_6H_4OCH_2CH_2$, $R_2 = C_6H_4OCH_2CH_2$; (IX) $R = C_6H_4CH_2CH_2$, $R_2 = C_6H_5$; (X) $R = C_6H_4CH_2CH_2$, $R_2 = C_6H_4NO_2$; (XI) $R = C_6H_4CH_2CH_2$, $R_2 = C_6H_5$; (XII) $R = C_6H_4CH_2CH_2$, $R_2 = C_6H_4NO_2$.

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1,4-bis(β -nitrovinyl)benzene reacted with two moles

7225

BOV/7 - 1-1-79

Table 6.

1	2	3			4			5
		C	H	N	C	H	N	
(I)	106—112°	60.30, 61.56	4.68	8.02	61.14	4.49	8.91	95
(II)	155—156	53.99, 53.54	4.22, 4.64	11.90, 11.90	53.48	3.65	11.7	85
(III)	106—109	59.04	4.93	8.12	59.30	4.68	8.14	86
(IV)	71—75	55.00, 55.46	3.93, 4.12	9.32, 9.45	55.26	3.58	9.21	54
(V)	113—115	52.54, 52.49	4.36, 4.71	8.78, 8.91	52.16	4.38	8.70	62
(VI)	106—112	59.05, 59.2	4.76, 4.85	8.05, 8.14	59.30	4.68	8.14	78
(VII)	148—148.5	52.67, 52.48	3.56, 3.79	11.12, 10.68	52.44	3.88	10.7	96

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Synthesis of α, γ -Dinitroketones

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(VIII)	125.5--128.5	57.92, 57.81	4.92, 4.79	7.65, 7.73	57.75	4.82	7.15	86
(IX)	94--97	—	—	8.40, 8.47	—	—	8.51	70
(X)	143--144	54.70, 54.91	3.86, 4.16	11.37, 11.29	54.69	4.05	11.26	81.5
(XI)	114--117	56.81, 59.81	3.84, 4.14	8.02, 7.89	56.98	3.94	7.80	67.5
(XII)	155--155.5	59.87, 59.89	2.99, 3.39	10.49, 10.40	59.63	3.25	10.42	87.5
XIII	184--185	56.59, 56.40	4.82, 4.52	10.14, 10.18	56.73	4.03	10.18	85
(XIV)	187--187.5	55.11, 54.90	4.29, 4.36	9.01, 8.89	55.08	4.29	8.96	57

Ref. to Table C. (1) Compound; (2) mp; (3) d_4^{20} (g/ml); (4) Calculated (%); (5) Yield (%).

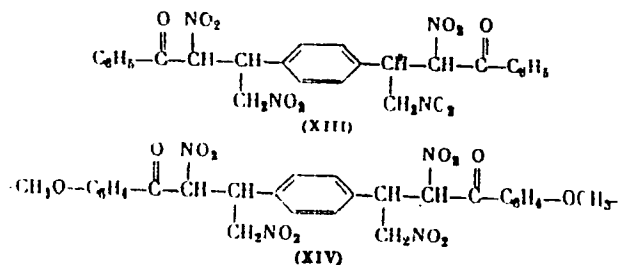
Card 4/5

Synthesis of α, β -Dinitroacetophenones

78286

SOV/79-30-3-40/69

of ω -nitroacetophenone and p-methoxy- ω -nitroacetophenone.



There is 1 table; and 7 references, 4 Soviet, 2 German, 1 U.K. The U.K. reference is: Arndt, F., Rose, J., J. Chem. Soc., 1935, 1.

ASSOCIATION: Gertsen Leningrad State Pedagogic Institute (Leningradskiy gosudarstvennyy pedagogicheskiy Institut imeni Gertsena)

SUBMITTED: April 15, 1959

Card 5/5

ZONIS, E.S.; PEREKALIN, V.V.

Synthesis of dinitrodienes. Zhur. prikl. khim. 33 no.6:1427-1428
Je '60. (MIRA 13:8)

(Hexadiene)
(Butane)

(Malonic acid)
(Aldehydes)

BOBOVICH, Ya.S.; PEREKALIN, V.V.; SOPOVA, A.S.

Determination of the structure of some dihydrofuran derivatives by means of Raman spectra. Dokl. AN SSSR 134 no.5:1083-1084 O '60.
(MIEA 13:10)

1. Leningradskiy gosudarstvennyy pedagogicheskiy institut im. A.I. Gertsena. Predstavleno akademikom A.N.Tereninym.
(Furan--Spectra)

PHASE I BOOK EXPLOITATION SOV/5808

Perekalin, Vsevolod Vasil'yevich

Nepredel'nyye nitrosoyedineniya (Unsaturated Nitro Compounds)
Leningrad, Goskhimizdat, 1961. 335 p. Errata slip inserted.
4500 copies printed.

Ed.: S. A. Zonis; Tech. Ed.: T. A. Pomkina.

PURPOSE : This book is intended for organic chemists, scientists of research institutes, instructors, and students in special chemistry schools.

COVERAGE: The book describes methods of synthesis, the nature of chemical conversions, and the practical application of unsaturated nitro compounds. The importance of these compounds as fungicides, insecticides, and medicinal substances is pointed out along with their value as monomers which, due to their easy polymerizability, can be converted to high polymer products. The role of unsaturated nitro compounds as initial products for

Card 1~~4~~6

SOPOVA, A.S.; PEREKALIN, V.V.; BOBOVICH, Ya.S.

Synthesis of dihydrofuran derivatives. Zhur.ob.khim. 31. no.5:
1528-1532 My '61. (MIRA 14:5)

1. Leningradskiy pedagogicheskiy institut imeni A.I.Gertsena.
(Furan)

ABRAMOVICH, T.I.; GRIGEROV, I.P.; PERIKALIN, V.V.

Isotopic hydrogen exchange in connection with the tendency toward
condensation reactions. Zhur.ob.khim. 31 no.6:1962-1968 Je '61.
(MIRA 14:6)

1. Institut fizicheskoy khimii im. I.V.Pisarzhevskogo AN Ukrainskoy
SSR i Leningradskiy pedagogicheskiy institut imeni A.I.Gertsena.
(Condensation products (Chemistry)) (Deuterium)

VASIL'YEVA, V.N.; PEREKALIN, V.V.; VASIL'YEV, V.G.

Study of the structure of unsaturated nitro compounds by the
method of dipole moments. Zhur.ob.khim. 31 no.7:2171-2175 J1
'61. (MIRA 14:7)

1. Leningradskiy gosudarstvennyy pedagogicheskiy institut imeni
A.I. Gertsena i Fiziko-khimicheskiy institut imeni L.Ya. Karpova.
(Nitro compounds)

VASIL'YEVA, V.N.; PEREKALIN, V.V.; VASIL'YEV, V.G.

Dipole moment study of the effect of steric factors on conjugation
in the molecules of unsaturated nitro compounds. Zhur.ob.khim.
31 no.7:2175-2178 J1 '61. (MIRA 14:7)

1. Leningradskiy gosudarstvennyy pedagogicheskiy institut imeni
A.I. Gertsena i Fiziko-khimicheskiy institut imeni L.Ya. Karpova.
(Nitro compounds—Dipole moments)

ZONIS, E.S.; LERNER, O.M.; PEREKALIN, V.V.

Synthesis of dinitrotrienes. Zhur.prikl.khim. 34 no.3:711-712 Mr
'61. (MIRA 14:5)

(Olefins)

20357

S/020/61/136/005/015/032
B103/B208

53610

2209, 1153, 1375

AUTHORS:

Baskov, Yu. V. and Perekalin, V. V.

TITLE:

Synthesis and chemistry of allyl nitrite

PERIODICAL:

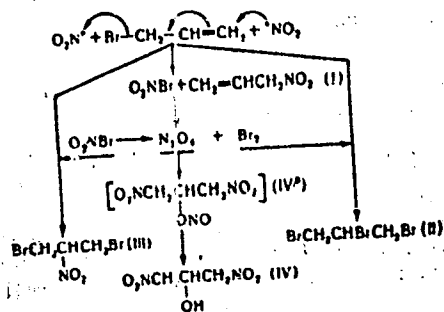
Doklady Akademii nauk SSSR, v. 136, no. 5, 1961, 1075-1078

TEXT: The authors obtained a surprisingly good yield of allyl nitrite (I) in the nitration of allyl iodide and allyl bromide with nitrogen tetroxide (N_2O_4) in ether at $-20^\circ C$. The resultant nitrile iodide and nitrile bromide are converted to more stable N_2O_4 , and to molecular iodine and bromine. Contrary to iodine, bromine brominates the initial allyl bromide during its formation, giving 1,2,3-tribromopropane (II). The appearance of 2-nitro-1,3-dibromopropane (III) is explained by nitro-halogenation of allyl bromide by nitrile bromide. If the reaction temperature is increased in the case of allyl iodide and allyl bromide, reaction of the resultant (I) with N_2O_4 leads to the synthesis of 1,3-dinitro propanol-2 (IV) which is considered to be a secondary

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Synthesis and chemistry of ...

nitration product. The chlorine-carbon bond in the allyl chloride is very stable, and the halogen is therefore not replaced in this case by the nitro group.



Accordingly, only 1-nitro-3-chloro propanol-2 (V) results from allyl chloride and no (I). The authors studied the chemical conversions of (I), and obtained substances which permitted the structure of some

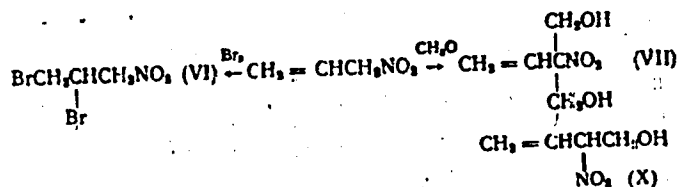
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20357

S/020/61/136/005/015/032
B103/B208

Synthesis and chemistry of ...

reaction products of allyl bromide and allyl iodide with N_2O_4 to be clarified. The absence of a pronounced mutual influence of the double bond and the nitro group (Ref. 5) suggests a combination of the properties of ethylene and nitro-ethane in (I). For this reason, the nitration of (I) by N_2O_4 yielded (IV). (IV) was isolated as a result of the hydrolysis of the original nitrate (IVa). Bromination yielded the only possible 1-nitro-2,3-dibromopropane (VI) which is isomeric to (III), and therefore has the structure assumed by the authors. Condensation with formaldehyde gives 2-nitro-2-methylol buten-3-ol-1 (VII) which was characterized by diacetate (VIII), and dibromide (IX).



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S/020/61/136/005/015/032
B103/B208

Synthesis and chemistry of ...

(VII) was converted to 3-nitro-buten-1-ol-4 (X) by splitting off the methylol group (in agreement with Ref. 6), which is an initial product in the synthesis of 2-nitro-butadiene-1,3. The authors emphasize that the above reaction is the first example of the synthesis of nitrogen compounds by the substituting nitration of allyl halogen derivatives by N_2O_4 . They assume that its mechanism may be explained by a conjugate allyl rearrangement under participation of allyl iodide, allyl bromide, and N_2O_4 . There are 6 figures and 6 references: 2 Soviet-bloc and 3 non-Soviet-bloc.

ASSOCIATION: Leningradskiy pedagogicheskiy institut im. A. I. Gertsena
(Leningrad Pedagogical Institute imeni A. I. Gertsen)

PRESENTED: September 1, 1960, by M. I. Kabachnik, Academician

SUBMITTED: September 1, 1960

Card 4/4

573230

11.2215

24044

S/020/61/138/003/016/017

B103/B208

AUTHORS: Sokolov, V. N., Poddubnyy, I. Ya., Perekalin, V. V., and Yevdokimov, V. F.

TITLE: Polymerization of nitroethylene under the action of γ -radiation

PERIODICAL: Doklady Akademii nauk SSSR, v. 138, no. 3, 1961, 619-620

TEXT: The authors devised methods for the industrial production of high-molecular nitroethylene under the action of γ -radiation since in this case products are obtained which are as pure as the initial monomers. Other methods with initiator and solvent yielded, so far only powdery products contaminated by initiator and solvent. Co^{60} was used as radiation source, the apparatus is described by A. Kh. Breger et al. (Ref. 9: *Deystviye ioniziruyushchikh izlucheniye na neorganicheskiye i organicheskiye polimernyye sistemy* (Effect of ionizing radiation on inorganic and organic polymer systems), Izd. AN SSSR, 1958). The initial nitroethylene was obtained by dehydration of 1-nitro-ethanol-2 with phthalic anhydride. Fractions with a boiling point of $36^{\circ}\text{C}/100$ mm Hg were isolated from the monomer by

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X

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S/020/61/138/003/016/017
B103/B208

Polymerization of nitroethylene ...

repeated fractionation. Hot nitrogen was bubbled through glass ampuls which were then filled with freshly distilled nitroethylene. The occluded atmospheric oxygen was removed by the usual freezing up and melting. The ampuls sealed in vacuo were irradiated at 20°C, and the monomer was distilled off in vacuo after opening. At the beginning of irradiation (dose $1 \cdot 10^6$ r), a turbidity was observed in the monomer which had hitherto been as clear as water. At a dose of $5 \cdot 10^6$ r a white precipitate results which is identical with the polymer resulting under the action of organic bases. On further irradiation, the pasty monomer-polymer mixture is converted to a transparent, pale-yellow polymer block. This is apparently related to secondary addition reactions of growing polymer chains to the polymer already formed, and is accompanied by an increase of its molecular weight. At doses > 0.3 Mr/hr no block polymer is formed. In this case the polymer remains powdery up to a 100% conversion, and turns light-brown. The formation of the block polymer being a very complicated physico-chemical process depending on many factors, a powder is formed in some cases even with a 100% conversion. The polymerization of partly

Card 2/5

Polymerization of nitroethylene ...

24044
S/020/61/138/003/016/017
B103/B208

polymerized samples continues also after irradiation is finished. This suggests the formation of rather long-live polymer radicals under the action of γ -radiation (Fig. 2). Also in this case block-poly-nitroethylene results. The polymerization is inhibited by hydroquinone and oxygen which confirms the radical nature of this process. The polymer is insoluble in common solvents, well soluble in N,N-dimethyl formamide. Its intrinsic viscosity in this solvent is 0.38 which corresponds to a molecular weight of 38,000. Its density is d_{20} 1.535, the decomposition temperature 150°C.

No denitrification ($-\text{CH}_2 - \text{CHNO}_2$)_n takes place during irradiation. The crystalline phase is absent (X-ray data by S. G. Strunskiy). An intense narrow halo and a weak broad halo correspond to the parameters of the

short-range order 5.15 Å and 3.73 Å. Under the action of γ -radiation nitroethylene may be copolymerized with other unsaturated nitro compounds such as 1,4-dinitro-butadiene-1,3. There are 3 figures and 9 references: 3 Soviet-bloc and 6 non-Soviet-bloc. The two most important references to English-language publications read as follows: Ref. 4: D. Vofsi, A. Katchalsky. J. Polym. Sci., 26, 127 (1957); Ref. 7: G. Buckley,

Card 3/5

Polymerization of nitroethylene ...

24044
S/020/61/138/003/016/017
B103/B208

C. Scaife. Brit. Pat. 595282, 1947; Chem. Abstr., 42, 37775 (1948).

PRESENTED: December 20, 1960, by N. N. Semenov, Academician

SUBMITTED: December 17, 1960

Card 4/5

BOBOVICH, Ya.S.; KVITKO, S.M.; PEREKALIN, V.V.

Study of the structure of nitroaminobutadiene derivatives
by means of Raman spectroscopy. Dokl. AN SSSR 139 no.6:1392-
1395 Ag '61. (MIRA 14:8)

1. Predstavleno akademikom A.N.Tereninym.
(Butadiene--Spectra)

VASIL'YEVA, V.N.; PEREKALIN, V.V.; VASIL'YEV, V.G.

Method of dipole moments used for studying the structure of unsaturated nitro compounds. Dokl. AN SSSR 141 no.3:620-623 1961.
(MIRA 14:11)

1. Predstavleno akademikom A.N. Frumkinym.
(Nitro compounds--dipole moments)

KVITKO, S.M.; PEREKALIN, V.V.

Synthesis of nitroaminobutadiene derivatives. Zhur. ob. khim. 32 no.1:
144-150 Ja '62. (MIRA 15:2)

1. Leningradskiy pedagogicheskiy institut imeni A.I.Gertsena.
(Butadiene)

VASIL'YEVA, V.N.; KVITKO, S.M.; PEREKALIN, V.V.

Study of the structure of derivatives of nitroaminobutadiene by the
method of dipole moments. Zhur.ob.khim. 32 no.6:1768-1771. Je '62.
(MIRA 15:6)

1. Leningradskiy gosudarstvennyy pedagogicheskiy institut im. A.I.
Gertsena.

(Butadiene---Dipole moments)

BASROV, Yu.V.; PEREKALIN, V.V.

Isomerization of 1-nitro-2-propene. Zhur.ob.khim. 32 no.9:3106
S '62. (MIRA 15:9)

1. Leningradskiy pedagogicheskiy institut imeni A.I. Gertsena.
(Propene)

KVITKO, S.M.; PEREKALIN, V.V.

Synthesis of nitropentadiene derivatives. Zhur.ob.khim.
32 no.10:3298-3301 0 '62. (MIRA 15:11)

1. Leningradskiy gosudarstvennyy pedagogicheskiy institut
imeni A.I. Gertsena.
(Pentadiene)

35731

S/020/62/143/002/015/022
B145/B138

117211

AUTHORS: Kvitko, S. M., Perekalin, V. V., Vasil'yeva, V. N.,
Bobovich, Ya. S., and Slovokhotova, N. A.

TITLE: Synthesis and structure of nitrobutadiene derivatives

PERIODICAL: Akademiya nauk SSSR. Doklady, v. 143, no. 2, 1962, 345 - 347

TEXT: Some nitrobutadiene and nitraminobutadiene derivatives were synthesized, and their structure was examined, in order to establish the effect of the chemical structure of nitralkenes and nitralkenedienes upon their polymerizability. The reaction scheme indicates the synthesis course as well as the products obtained. This is the first case of a C-chain condensation with malonic acid aldehyde. A ketimino - enamino tautomerism can be dismissed for products of the Knoevenagel condensation. It was not possible to alkylate nor to acylate the amino and nitraminobutadienes. The spatial structure of compounds II to X (see diagram) was examined by Raman spectra and by measuring the dipole moments. III and IV (compound IV is not indicated in the diagram; its structure is the same as that of

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X

S/020/62/143/002/015/022
B145/B138

Synthesis and structure ...

VII - IX, except that there is $=NC_6H_5$ instead of $=CRR'$) exhibit intramolecular H bonds; the nitro group is in cis-position with respect to the amino group. Compounds V - VIII do not possess a plane structure. The nitro group (at the C-N bond) as well as the vinylidene residue (at the C-C bond of the butadiene grouping) are deflected here. The H bonds are also weakened thereby, which results in a displacement of the fully symmetric vibration band of the nitro group toward shorter wavelengths ($\nu = 1350$). IX exhibits a high dipole moment (7.5 D), which is explained by assuming a structure in which the dipole moments of the two nitrile groups add. A characteristic of the Raman spectra of nitraminobutadienes was found to be the splitting of the fully symmetric vibration of the nitro group, which may be caused by the intramolecular H bonds or the Fermi resonance. When examining the concentration dependence no redistribution of intensities was observed. Hence, the splitting cannot be caused by intermolecular H bonds. The intensity of the nitro-group bands is considerably higher in aromatic derivatives (IV:140) than in aliphatic ones (VI:12). This circumstance indicates the inclusion of an aromatic ring in the conjugation through the amino group. The low intensity of double bond

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vibration is explained by the weakening effect of the H ring upon the double bond. The vibrational intensities in double bond and antisymmetric $-C_6H_5$ are relatively high for IX and X (IX: 90 and 80, X: 20 and 75 ÷ 45, respectively). Evidently, a conjugation in IX, that involves all π - electrons, is of greater advantage from the energy viewpoint than would be a conjugation, wherein only the H ring participates. In X, by contrast, the possibility that an H ring may form is lacking altogether. There are 1 table and 5 references: 4 Soviet and 1 non-Soviet. The reference to the English-language publication reads as follows: D. Hathuay, M. Flett, Trans. Farad. Soc., 45, 818 (1949).

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X

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